PB-248 103

COLLECTION EFFICIENCY STUDY OF THE PROPOSED METHOD 13 SAMPLING TRAIN

Walter Smith

Entropy Environmentalists, Incorporated

Prepared for:

Environmental Sciences Research Laboratory

September 1975

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PB 248 103

EPA-600/2-75-052
September 1975

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EPA-600/2-75-052
September 1975

COLLECTION EFFICIENCY STUDY OF THE PROPOSED METHOD 13 SAMPLING TRAIN

by

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Contract No. 68-02-1792

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ABSTRACT

This report summarizes the testing undertaken to determine the collection efficiency of the proposed Method 13 sampling train. During the course of the testing, an investigation into the accuracy and precision of the two Method 13 analytical techniques for determining fluorides was necessitated.

After constructing a source capable of producing known concentrations of fluorides, tests were run on this source at four different concentrations and two different flow rates.

Analysis was performed initially using the Method 13(A) Spadns Zirconium Lake colorimetric method. Erratic and non-reproducible data prompted analyses omitting the distillation procedure, and analyses using the Method 13(B), fluoride specific ion electrode technique.

Results of these analyses demonstrated that the accuracy and precision of the fluoride specific ion electrode method was better than the Spadns Zirconium Lake colorimetric method, the latter showing a positive bias.

From all the data collected, conclusions can be drawn only as to the collection efficiency of the sampling train using fluoride concentrations in the range of 6 to 118 parts per million, and at sampling rates of 3/4 and 1 cubic foot per minute. The results indicated a collection efficiency of 99% on gaseous hydrogen fluoride (HF).

This report was submitted in fulfillment of Contract Number 68-02-1792 by Entropy Environmentalists, Inc., under the sponsorship of the Environmental Protection Agency. Work was completed as of September 1975.

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO.	2.	3. RECIPIENT'S ACCESSION NO.
EPA-600/2-75-052		

4. TITLE AND SUBTITLE	5. REPORT DATE
COLLECTION EFFICIENCY STUDY OF THE PROPOSED METHOD 13 SAMPLING TRAIN	September 1975 (Approval Date)
	6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S)	8. PERFORMING ORGANIZATION REPORT NO.
Walter Smith	
9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. PROGRAM ELEMENT NO.
Entropy Environmentalists, Inc. Research Triangle Park, N.C. 27709	1AA010
	11. CONTRACT/GRANT NO.
	68-02-1792
12. SPONSORING AGENCY NAME AND ADDRESS	13. TYPE OF REPORT AND PERIOD COVERED
Environmental Sciences Research Laboratory Office of Research and Development U. S. Environmental Protection Agency Research Triangle Park, N. C. 27711	Final
	14. SPONSORING AGENCY CODE
	EPA-ORD

15. SUPPLEMENTARY NOTES

16. ABSTRACT

This report summarizes testing undertaken to determine the collection efficiency of the proposed Method 13 sampling train. The efficiency of the train in the concentration range 6-118 ppm and at a sampling rate of 3/4 to 1 cfm was found to be 99+%. Laboratory analyses of samples using the SPADNS and the SIE techniques for fluoride determination showed that the SPADNS method is less precise and accurate than the specific ion electrode. It was also determined that the distillation step in the analytical procedure resulted in erratic and non-reproducible results. Analyses requiring this distillation step must be watched carefully to avoid carry over of potential interfering ions.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Collecting methods		14B
Evaluation		13B
Efficiency		7B
Air pollution		7D
Fluorides		
Chemical Analysis		

PRICES SUBJECT TO CHANGE

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS (This Report)	21. NO. OF PAGES
RELEASE TO PUBLIC	UNCLASSIFIED	34
1.	20. SECURITY CLASS (This page)	22. PRICE
	UNCLASSIFIED	4.00 -225

EPA Form 2220-1 (9-73)

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ACKNOWLEDGEMENTS

The construction and operation of the experiment equipment was performed by Thomas J. Porzio, Emil Stewart, and Joseph Schiappa of EEI. The report was written by Emil Stewart and Walter S. Smith. The project was directed by H. M. Barnes of EPA. Mr. Fred Grainger of Grainger Laboratories, Inc. provided the analytical portion of the data.

Section I
CONCLUSIONS

It was found that the collection efficiency of the proposed Method 13 sampling train, with the filter between the third and fourth impingers, was approximately 99%. Analysis using the fluoride specific ion electrode technique gave an average collection efficiency of 97.6% while the Spadns colorimetric method resulted in a 100.4% collection efficiency. Both analyses were done without using the distillation step, a procedure which gave erratic and non-reproducible results.

The results from the specific ion analytical method are more reproducible than those from the Spadns method. For the specific ion, the standard deviation was 4.5% of the mean on replicate average samples, and 4.3% on replicate analyses. The Spadns analyses resulted in a standard deviation of 6.7% of the mean on replicate average samples, and 7.4% on replicate analyses.

The Spadns method has several serious deficiencies, which became apparent during the project. The colorimetric determination is affected by the presence of sulfate ion, giving a positive bias, and the amount of sulfate carry-over is a function of distillation temperature. Even if an attempt is made to compensate for the sulfate interference in the results, inaccuracies will result since a temperature change of 1°-3° C will cause significant biases. Since there is a limit on the amount of fluoride ion that can be distilled (0.6 mg), samples with high fluoride concentrations produce larger errors, due to a multiplication of biases.

An additional problem with the Spadns method is the one to two hour lag time before the samples reach a constant absorbance. Exposure to light apparently does not affect the results over short time spans, nor does the temperature of the samples during preparation. After preparation, however, the samples temperature must be close to that of the spectrophotometer cell chamber, or the absorbance will change while the readings are being taken.

The specific ion electrode method is not affected by sulfate ion concentrations lower than 1900 parts per million, and consequently distillations can be made at temperatures as high as 190° C. Calibration of the electrodes should be made at the beginning and end of sample runs, since there can be shifts in the calibration curve.

An experienced analytical chemist could make the Spadns method reproducible within acceptable limits if he were exacting in distillation temperatures, the time for color development, the temperature of the cell, etc. However, the method is by no means definitive, while the specific ion technique does approach a "cookbook" method.

Section II
RECOMMENDATIONS

A further investigation into Method 13 is indicated from the results obtained from the project, if it is necessary to use the method on sources which have concentrations and required sampling rates outside the ranges covered in this evaluation. It could then be determined if the efficiency of collection is affected by concentrations and flow rates different from those specified in the contract.

The sampling train efficiency could be checked by running a number of tests using high and low fluoride concentrations and flow rates. This will determine if the data collected from fluoride specific ion electrode method indicates a decrease in efficiency with high or low concentrations and flow rates, or if the specific ion has a negative bias at certain concentrations and flow rates.

It should be determined if greater quantities of fluoride can be distilled at higher temperatures. The specific ion method is not affected by sulfate at the 1900 ppm level, although it may be possible to distill larger quantities of fluoride at higher temperatures. Large errors resulting from sample dilutions could be avoided by distillation of entire samples.

Also it is recommended that a study be conducted to determine if the acetone and filter paper from actual field source sampling could be treated with a solution of sodium hydroxide instead of calcium oxide. Calcium fluoride is not soluble under acid conditions and may precipitate during the sulfuric acid washing of crucibles.

The Spadns method should not be used on aliquots from high fluoride concentrations. The resulting sulfate error from multiple dilutions can be very large. Thermometers should be calibrated at the actual immersion depth existing at the end point of the distillation, since a 1-3 degrees centigrade variation will result in large changes in sulfate concentration of distillates.

Section III
INTRODUCTION

Of the sources with New Source Performance Standards currently under development by EPA, there are two requiring fluoride emission measurements: phosphate fertilizer plants and aluminum reduction plants.

Fluoride determination measurements will be required for total fluorides (both gaseous and particulate) in stack gas samples. A combination particulate-gas sampling train consisting of a heated probe, glass filter holder and modified Greenburg-Smith impingers, prefilled with distilled water, has been proposed to collect the fluorides. Whatman #1 filter paper, placed either between the probe and first impinger or between the third and fourth impingers, collects the particulate fluorides. The gaseous fluorides can react with a hot glass probe to form silicon tetrafluoride which then, in contact with the water in the impingers, forms soluble fluosilicic acid and slightly soluble orthosilicic acid. Later, using either the Spadns Zirconium Lake colorimetric or fluoride specific ion electrode method, the fluorides can be determined.

Sampling efficiency problems for fluoride emissions from stationary sources have been reported for low concentrations of fluoride (10 ppm by volume).

Efficiencies of 50-60% have been found, although contradictory efficiencies have also been reported at different concentrations and flow rates.

Since the establishment of emissions standards is affected by the collection efficiency of the sampling technique and sample analysis, it is important that the question of efficiency of the proposed sampling train and analysis be answered before the final promulgation of the fluoride methods and standards.

Testing was done in June, 1975 by Entropy Environmentalists, Inc. (EEI) to determine the fluoride collection efficiency of the proposed

sampling train. Table I below presents the conditions specified for the thirty-three tests made on a source with various known fluoride concentrations.

Table 1

SUMMARY OF TEST CONDITIONS

Nominal Flow Rate CFM	HF Concentrations PPM by Volume Specified	Actual	Number of Tests
1	0	0	1 (Blank)
3/4	0-10	6	6
	25-35	30	4
	55-70	62	3
	95-115	103	3
1	0-10	6	6
	25-35	34	4
	55-70	64	3
	95-115	112	3

Initially, analysis was performed using the Spadns Zirconium Lake colorimetric method following distillation of the samples. However, due to the erratic and non-reproducible results, the analytical procedure was modified, with the approval of the project officer, to use both of the Method 13 analytical techniques (Spadns and specific ion) without prior distillation. The distillation step was determined to be unnecessary for these tests since only HF and water could be present in the sample (no interferences and no water-insoluble fluorides).

The design and fabrication of the testing equipment are detailed in Section IV. Section V describes the actual testing and analytical procedures, while in Section VI, the results are critically evaluated and recorded. The appendices are in Section VII and contain the tabulated raw analytical data of the collection efficiency experiments.

Section IV
EXPERIMENT DESIGN AND CONSTRUCTION

In order to determine the efficiency of collection of fluorides of the proposed Method 13 sampling train, the amount of fluorides produced by the source must be accurately known. This can be accomplished by generating gaseous HF in precisely known concentrations by volatizing aqueous HF solutions with heated air. The heated air stream would then be used as a source of known fluoride concentration.

EEI originally proposed that the above was to be achieved by dissolving HF into distilled water at the appropriate concentrations, using the equipment set-up shown in Figure 1. The aqueous HF solution was to be metered by a Nalgene titration burette fitted into one arm of a Teflon "T" fitting. The air stream was to be supplied by a pressurized "Zero Air" cylinder, which forces the air through a Tedlar bag (acting as a surge tank), and then into the Teflon "T" fitting where the aqueous HF is introduced. From the "T" fitting, the aqueous HF was to be carried by the "clean air" through a coil of Teflon tubing placed in an insulated, thermostatically controlled heating chamber. This chamber was to have heated the air stream and have evaporated the aqueous HF so that gaseous HF would have emerged from the heating chamber and passed into the proposed Method 13 sampling train. The HF levels being sampled would have been regulated by the concentration and rate of aqueous HF metered into the air stream.

At the start of the experiment construction, EEI discovered that the heating chamber could not evaporate all of the aqueous HF before the "clean air" stream entered the sampling train, due to the size of the HF drops.

By reducing the size of the aqueous HF drops, EEI hoped to alleviate the evaporation problem; this was achieved by creating an orifice aspirator at the point where the burette tip was placed in the clean air stream. The prohibitive problem encountered with this design was the high pressure drop needed to aspirate the HF drops.

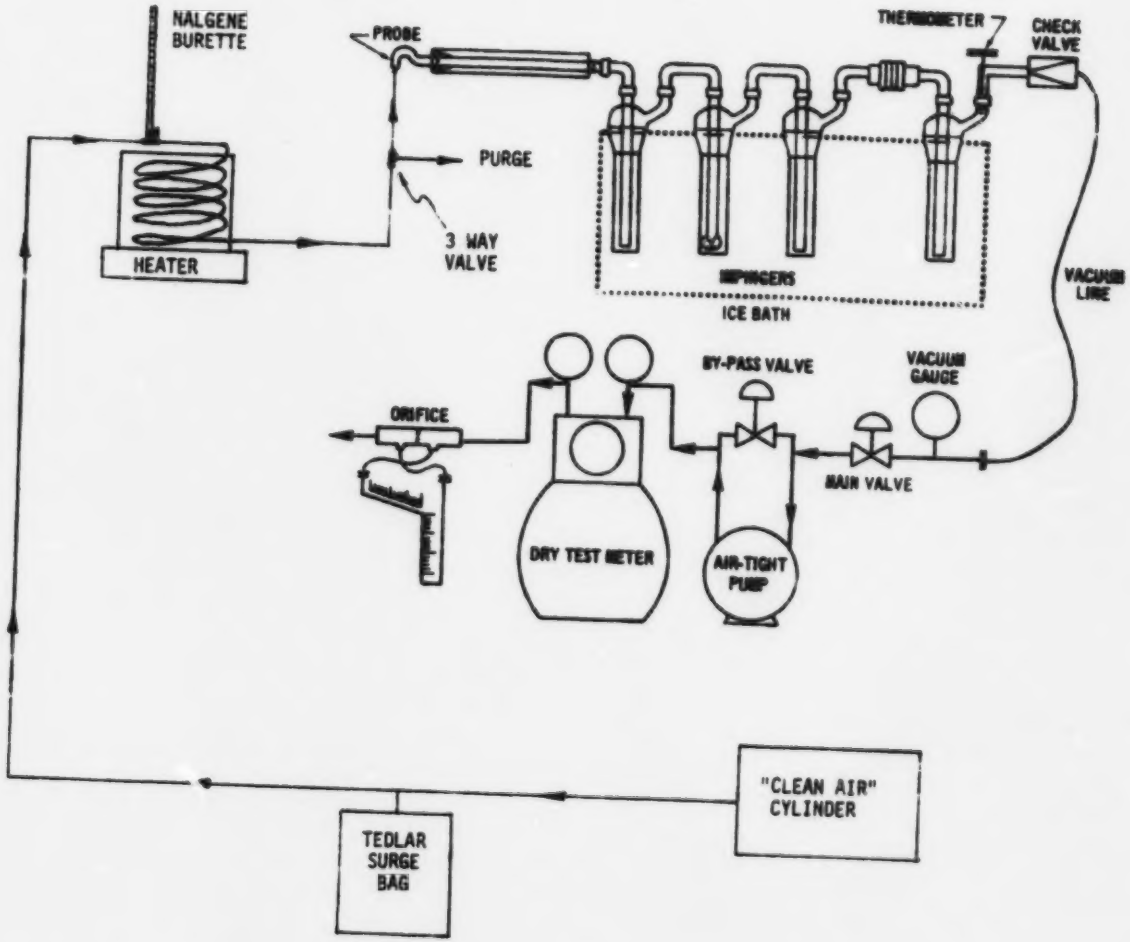

Figure 1. PROPOSED HF GENERATION AND SAMPLING SYSTEM

Finally the design shown in Figure 2 was arrived at. First an air preheater was placed between the Tedlar surge bag and the Teflon "T" fitting. The preheater consisted of copper tubing immersed in a hot oil bath kept at temperatures between 300° and 360° F by an electric hot plate. Also, the Teflon tubing coil was taken out of the heating chamber and placed in a boiling water bath so that better heat conduction to the Teflon tubing would be achieved. With this source of known fluoride concentrations, EEI was ready to commence testing.

The proposed sampling train to be tested consisted of a stainless steel nozzle with a sharp, tapered leading edge. The probe liner was made of pyrex, with a heating system capable of maintaining 250° F. The filter holder was assembled with Whatman #1 filter paper and placed between the third and fourth impingers. Distilled water was placed in the first and second impingers, and silica gel was placed in the fourth. All of the impingers were of the modified Greenburg-Smith construction, except for the second which was a standard Greenburg-Smith design. The metering system, connected to the sampling box by an umbilical line, consisted of a vacuum gauge, leak-free pump, thermometers, dry gas meter, and related equipment necessary to maintain an isokinetic sampling rate and to determine sample volume.

With this equipment, the following regimen was followed.

Table 2. TEST PROTOCOL

Specified Conc. Range, PPM	Number of Tests @ Each CFM	Burette Solution mg HF/ml	3/4 CFM		1 CFM	
			mg HF/hr	ml/hr	mg HF/hr	ml/hr
0-10	6	0.18	5.4	30	7.2	40
25-35	4	1.11	33.3	30	44.4	40
55-70	3	1.76	63.4	36	82.7	47
95-115	3	3.04	109.4	36	142.9	47

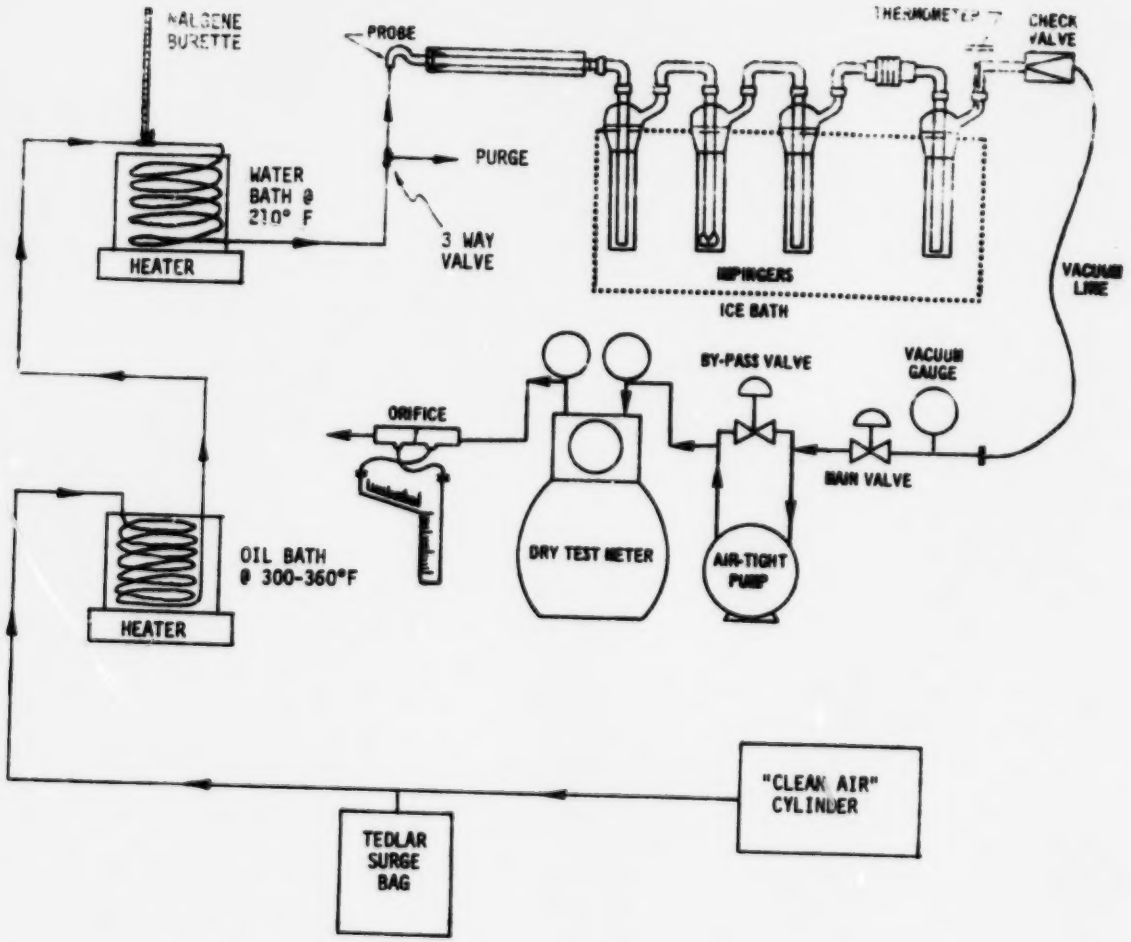

Figure 2. HF GENERATION AND SAMPLING SYSTEM

Table 2 gives the volume of the given concentration of aqueous HF burette solution needed to be introduced into the heated clean air stream, to achieve the parts per million concentration specified by the contract. The concentration of the burette HF solution was determined on several runs by the fluoride specific ion electorde method and by an acid-base titration, with the results agreeing within one percent.

The equipment was set up and connected as shown in Figure 2, with the heaters in operation. Aqueous HF solution was placed into the burette. The evaporation system was started, and once the rate had stabilized, readings were taken on the burette. The sampling train (after being leak tested) was connected to the evaporator system, and a stopwatch was started. At the end of a one-hour period, the sampling train was stopped and a final reading taken on the burette.

Clean-up consisted of washing all sample-exposed surfaces with distilled water, followed by an acetone washing. A sample of the burette solution was analyzed after each run, along with the samples. The proposed method of analysis was duplicate Spadns Zirconium Lake colorimetric analyses with distillation; however, the fluoride specific ion electrode technique was also employed, and the Spadns method was used without distillation.

Section V
TESTING OPERATION AND ANALYSIS PROCEDURES

Actual testing was done in the EEI building located on U. S. Highway 70 West, Raleigh, North Carolina. Testing started June 12, 1975, and continued until June 30, 1975. During this period thirty-three runs were made: thirty-two at the specified conditions and one as a control blank.

Table 3. TEST LOG

Test Number	Testing Date	Test Number	Testing Date
Blank (1)	6-12-75	14-16	6-24-75
2	6-13-75	17-19	6-25-75
3-5	6-17-75	20-24	6-26-75
6	6-18-75	25-28	6-26-75
7-9	6-19-75	29-30	6-28-75
10-13	6-20-75	31-33	6-30-75

A Spadns analysis was performed on the first twelve samples with the resulting efficiencies being well over 100%. The error seemed to originate from the distillation process, so the analyses were done without the distillation process. The distillation was not really necessary for these particular samples, since interferences should not have been present. After the thirty-three analyses were finished and the results reviewed, the samples were analyzed again using the fluoride specific ion electrode method, which gave better reproducibility.

The method for determining the amount of fluoride introduced into the sampling train was achieved as proposed. As described in Section IV, a gaseous fluoride sample was collected using the proposed Method 13 sampling train (with filter between the third and fourth impingers). The nozzle probe, filter impingers, and connectors were washed first with distilled water and then with acetone, generating two samples for each test.

After transferring the collected samples to appropriate containers, CaO was added to the water sample which formed a basic slurry. The water was then evaporated, NaOH added, and the sample fused. Warm water and sulfuric acid were used to rinse the sample into a flask. Here the analysis diverges from the specified methods, which call for a distillation process. Instead, the Spadns mixed reagent or the Tisab solution were added and readings taken, depending upon the technique being used. The spectrophotometer used was a Bausch and Lomb Model 700.

The acetone washings had CaO added to them, which did not disperse. To facilitate dispersion, water was added and the sample shaken vigorously, after which the acetone was evaporated. Again, the residue was fused with NaOH. The remaining steps are the same as with the water sample.

Section VI
DISCUSSION OF RESULTS

After the analysis of the first twelve samples, it was concluded that the quality of the results using the Spadns method following distillation was questionable, and that this method could not be used reliably for determining collection efficiency. Table 4 indicates that when the Spadns method is used following distillation, the results were an average of 18% higher than those analyzed without the distillation step.

Since the presence of sulfate ion gives a positive interference in the Spadns method, it was suspected that sulfates were being carried over in the distillation step from the sulfuric acid used in treating the samples. Tests were performed to determine the amount of carry-over and Figure 3 clearly demonstrates that the amount of sulfate ion carry-over is significant, while also a function of distillation temperature. Using the recommended distillation temperature of 180° C, and following all the other requirements and recommendations of the method (e.g., a maximum of 0.6 mg HF in the distillate), enough sulfate ion is carried over to give a positive bias equivalent to 0.07 mg HF/ml. This represents 10-15% of the HF concentration at the mid-range of the colorimeter scale.

Because of this sulfate interference, it was decided to determine the sampling efficiency by Spadns and specific ion methods without distillation and compare the precision of the two methods for paired data. Tables 5 and 6 show the data obtained. Each run was made from a different dilution series except in the case of Table 5 Spadns results.

A sample of the distillate from a 190° C distillation was prepared to contain 0.1 micromole of sodium fluoride, and this was analyzed along with a water sample containing 0.1 micromole of sodium fluoride, using the specific ion electrode method. The millivolt readings from the electrode on the two samples were identical, leading to the conclusion that the specific ion method is not as sensitive to the presence of sulfate ion as the Spadns method.

Table 4

COMPARISON OF AVERAGE FLUORIDE MEASURED BY SPADNS METHOD BEFORE AND AFTER DISTILLATION

Run Number	mg F Before Distillation	mg F After Distillation	mg F Difference	% Difference
1	0	0.92	0.92	-
2	5.89	5.22	-0.67	-11.4
3	6.23	5.53	-0.70	-11.2
4	6.26	5.83	-0.43	-0.69
5	5.29	5.38	0.09	1.7
6	6.46	7.38	0.92	14.2
7	4.47	7.38	2.91	65.1
8	7.07	9.23	2.16	30.6
9	7.01	9.34	2.33	33.2
10	7.04	8.92	1.88	26.7
11	8.33	9.92	1.59	19.1
12	7.23	9.92	2.69	37.2

From % difference

Mean = + 18.0%

Standard deviation = 29.0%

Table 5

COMPARISON OF DUPLICATE DATA BY THE SPADNS FLUORIDE METHOD WITHOUT DISTILLATION

Run Number	mg F Aliquot 1	mg F Aliquot 2	mg F Mean	mg F Difference	% Difference From Mean
1	0	0	0	0	0
2	7.26	4.51	5.89	2.75	23.3
3	7.38	5.13	6.23	2.25	18.1
4	7.38	5.13	6.26	2.25	18.0
5	5.66	4.92	5.29	0.74	7.0
6	6.77	6.15	6.46	0.62	4.8
7	4.42	4.51	4.47	0.09	1.0
8	7.38	6.76	7.07	0.62	4.4
9	7.38	6.64	7.01	0.74	5.3
10	7.78	6.70	7.04	0.69	4.9
11	8.61	8.05	8.33	0.55	3.3
12	7.38	7.07	7.23	0.31	2.1
13	7.26	6.76	7.01	0.50	3.6
14	36.9	30.8	33.9	6.10	9.0
15	35.9	30.4	33.2	5.60	8.4
16	32.3	30.4	31.4	2.00	3.2
17	35.1	30.3	32.7	4.80	7.3
18	50.4	49.8	50.1	0.60	0.6
19	46.1	43.1	44.6	3.00	3.4
20	36.9	40.6	38.8	3.70	4.8
21	33.8	40.0	36.9	6.20	8.4
22	66.4	58.4	62.4	8.00	6.4
23	67.7	67.0	67.4	0.70	0.5
24	61.5	65.8	63.7	4.30	3.4
25	83.6	84.9	84.3	1.30	0.8
26	80.0	83.0	81.5	3.00	1.8
27	86.1	86.1	86.1	0	0
28	110.7	109.5	110.1	1.2	0.5
29	110.7	108.9	109.8	1.8	0.8
30	110.7	110.7	110.7	0	0
31	145.1	156.4	150.8	11.3	3.7
32	141.5	155.2	148.4	13.7	4.6
33	146.4	156.4	151.4	10.0	3.3

FROM % DIFFERENCE

Standard Deviation = 7.4% from mean

Table 6

COMPARISION OF DUPLICATE DATA FOR FLUORIDE DETERMINATIONS BY THE SPECIFIC ION ELECTRODE METHOD

Run Number	mg F Aliquot 1	mg F Aliquot 2	mg F Mean	mg F Difference	% Difference From Mean
1	0	0	0	0	0
2	4.86	4.86	4.86	0	0
3	5.62	5.24	5.43	0.38	3.5
4	5.62	5.27	5.45	0.35	3.2
5	5.62	5.05	5.34	0.57	5.3
6	6.83	6.30	6.57	0.53	4.0
7	5.69	5.20	5.45	0.49	4.5
8	7.52	7.33	7.43	0.19	1.3
9	7.48	6.57	7.03	0.91	6.4
10	7.33	6.41	6.87	0.92	6.7
11	8.66	6.41	7.67	2.51	16.4
12	7.79	6.91	7.35	0.88	6.0
13	7.10	6.57	6.84	0.43	3.1
14	36.1	33.0	34.6	3.28	4.7
15	34.6	31.7	33.2	2.9	4.4
16	33.8	31.5	32.7	2.3	3.5
17	34.4	31.7	33.1	2.7	4.1
18	51.3	50.1	50.7	1.2	1.2
19	44.8	43.7	44.3	1.1	1.2
20	42.2	40.3	41.3	1.9	2.3
21	40.3	38.7	39.5	1.6	2.0
22	60.8	58.9	59.9	1.9	1.6
23	63.8	62.7	63.3	1.1	0.9
24	61.5	60.0	60.8	1.5	1.2
25	79.8	76.7	78.3	3.1	3.0
26	80.5	77.1	78.8	3.4	2.2
27	79.8	76.3	78.1	3.5	2.2
28	109.0	105.2	107.1	3.8	1.7
29	108.2	104.5	106.4	3.7	1.7
30	107.9	101.8	104.9	6.1	2.9
31	134.0	136.7	135.4	2.7	1.0
32	137.9	136.7	137.3	1.2	0.4
33	134.8	136.7	135.8	1.9	0.7

FROM % DIFFERENCE

Standard Deviation = 4.3% from mean

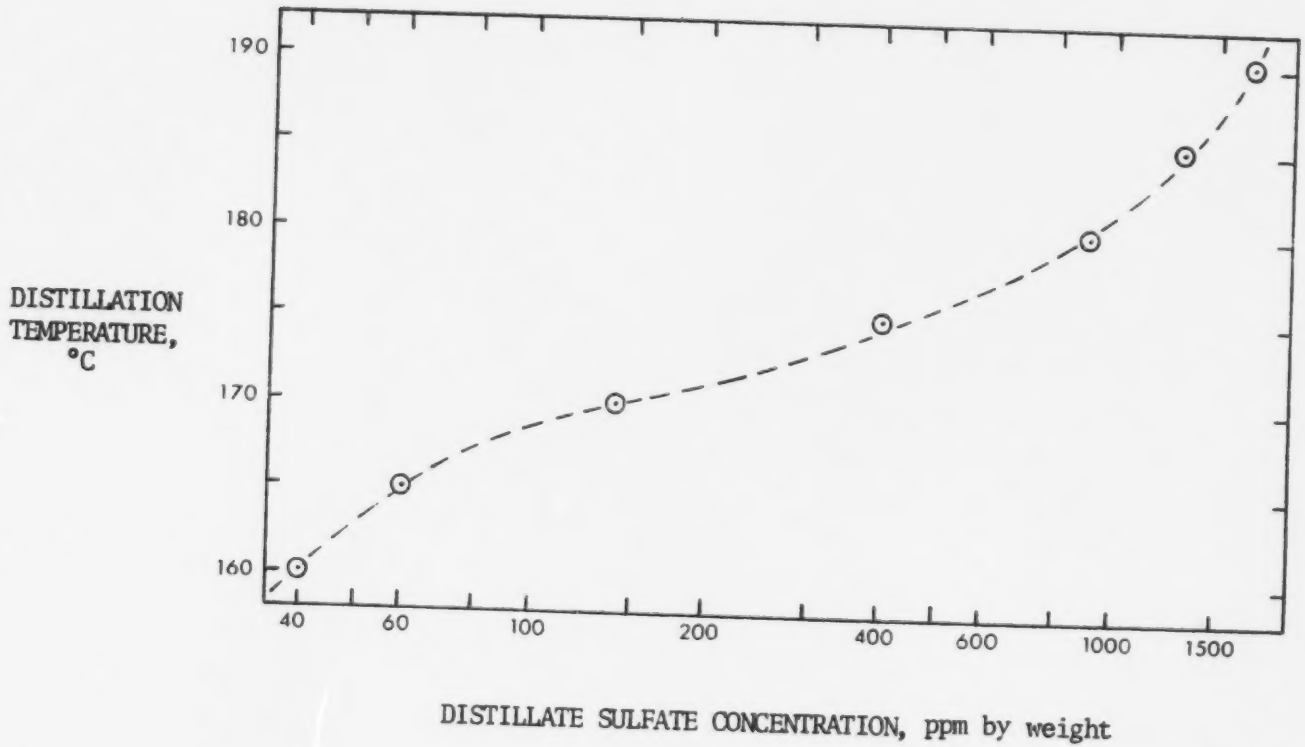

Figure 3. SULFATE CARRY-OVER DURING DISTILLATION

A series of samples containing 0.526 µg/ml of HF were prepared at different temperatures, and then cooled to ambient temperature to determine if temperature of preparation affected the results. Earlier erratic results were originally attributed to the Spadns solution being hot from sitting in the sunlight or being photo-sensitive. The results shown in Table 7 below were obtained at the indicated temperature of preparation in sunlight after conditioning to ambient temperature.

Table 7. Effect of Preparation Temperature on Absorbance.

Temperature of Preparation	Absorbance
20° C	0.840
22° C	0.835
24° C	0.835
26° C	0.835
28° C	0.840
30° C	0.840
32° C	0.840

The data yields a standard deviation of the fluoride concentration to be 1.7%, indicating that the temperature of preparation is not critical and that the reaction is not photo-sensitive.

A series of samples and standards were prepared at 20° C. The absorbance values increased during residence in the spectrophotometer over a period of several minutes, until they became constant at the temperature of the cell chamber (23° C). The change was a significant 0.005-0.02 absorbance units or an increase of 0.0739 µg/ml.

Calibration changes, although small, occurred between the start and finish of sample analyses by the specific ion method when analyzed at 24° C ± 0.25° C. The specific ion results are based on average calibration curves obtained from data at the beginning and end of sample runs.

Table 8. COLLECTION EFFICIENCY SUMMARY (WITHOUT DISTILLATION)

Run Number	Nominal Flow Rate cfm	Nominal Concentration ppm by volume	Efficiency - Spadns Method			Efficiency - Specific Ion Method		
			%	Average	Std. Dev.	%	Average	Std. Dev.
1	1	0	-	-	-	-	-	-
2	3/4	6	109.1	105.7	13.1	90.0	101.1	8.2
3			115.4			100.6		
4			115.9			100.9		
5			97.6			98.5		
6			113.5			115.5		
7			92.9			100.9		
8	1	6	98.2	98.4	1.2	103.2	97.5	4.5
9			97.4			97.6		
10			97.8			95.4		
11			99.3			91.4		
12			100.4			102.1		
13			97.4			95.0		
14	3/4	32	100.9	98.1	2.8	103.0	99.9	2.1
15			99.4			99.4		
16			94.3			98.2		
17			97.9			99.1		
18	1	32	97.7	94.0	5.6	98.8	97.1	2.7
19			99.1			98.4		
20			87.4			93.0		
21			91.6			98.0		
22	3/4	63	99.0	99.6	0.7	95.1	94.6	0.4
23			100.4			94.3		
24			99.4			94.9		
25	1	63	101.9	101.5	2.8	94.7	94.8	0.5
26			98.5			95.3		
27			104.1			94.4		
28	3/4	108	100.6	100.4	0.9	97.9	96.7	1.0
29			99.5			96.4		
30			101.2			95.9		
31	1	108	105.5	104.5	2.1	94.8	94.8	0.3
32			102.1			94.5		
33			105.9			95.0		
average				100.4	6.7		97.6	4.5

The data for the collection efficiency is shown in Attachments A and B of the Appendix, using the Spadns and specific ion electrode methods (without distillation) respectively. Summaries of this data are presented in Table 8 for the same respective methods. The efficiency appears to be 100% when compared by the Spadns method. However, as seen in Table 8, the efficiency determined using the specific ion electrode method is 98%.

The effect of varying flow rates and concentrations presented in Table 8 shows no apparent bias in the collection efficiency using the Spadns results. Results determined by the specific ion electrode method hint at a possible relationship among concentration, flow rate and collection efficiency. Whether the relationship exists, and whether it is caused by the analytical method or the sampling train, is not clear.

Section VII
APPENDIX

Attachment A

SAMPLING AND ANALYTICAL DATA, SPADNS METHOD WITHOUT DISTILLATION

Run Number	Orifice Pressure *in H2O*	Meter Pressure *in Hg*	Volume of Gas Metered *acf*	Meter Temp. *°R*	Volume of Gas Metered *dscf*	Gas Flow Rate *dscfm*	HF Solution Input		HF Input *mg*	HF Conc *ppm by volume*	HF Output *mg*	Collection Efficiency *%*
							Volume *ml*	Conc *mg/ml*				
Blank	3.7	29.53	59.84	574	55.02	0.92	40.0	0.00	0.00	0.0	0.00	-
2	1.6	29.50	44.05	557	41.48	0.69	30.0	0.18	5.40	5.83	5.89	109.1
3	1.6	29.63	44.87	557	42.44	0.71	30.0	0.18	5.40	5.70	6.23	115.4
4	1.6	29.63	44.01	559	41.48	0.69	30.0	0.18	5.40	5.83	6.26	115.9
5	1.6	29.63	44.93	563	42.04	0.70	30.1	0.18	5.42	5.78	5.29	97.6
6	1.6	29.68	45.08	558	42.63	0.71	31.6	0.18	5.69	5.98	6.46	113.5
7	1.6	29.70	44.05	551	42.22	0.70	30.0	0.18	5.40	5.73	4.47	82.8
8	2.9	29.70	61.42	560	58.10	0.97	40.0	0.18	7.20	5.55	7.07	98.2
9	2.9	29.70	61.62	565	57.78	0.96	40.0	0.18	7.20	5.58	7.01	97.4
10	2.9	29.58	60.36	555	57.38	0.96	40.0	0.18	7.20	5.62	7.04	97.8
11	2.9	29.58	61.04	562	57.31	0.96	46.6	0.18	8.39	6.56	8.33	99.3
12	2.9	29.58	62.06	565	57.96	0.97	40.0	0.18	7.20	5.57	7.23	100.4
13	2.9	29.58	61.07	567	56.83	0.95	40.0	0.18	7.20	5.68	7.01	97.4
14	2.4	29.83	54.56	555	52.24	0.87	30.3	1.11	33.6	28.8	33.9	100.9
15	2.4	29.83	55.25	565	51.97	0.87	30.1	1.11	33.4	28.8	33.2	99.4
16	2.1	29.83	51.45	567	48.19	0.80	30.0	1.11	33.3	31.4	31.4	94.3
17	2.0	29.76	48.48	552	46.52	0.78	30.1	1.11	33.4	32.2	32.7	97.9

Attachment A, Continued

Run Number	Orifice Pressure *in H2O*	Meter Pressure *in Hg*	Volume of Gas Metered *acf*	Meter Temp. ^{o}R	Volume of Gas Metered *dscf*	Gas Flow Rate *dscfm*	HF Solution Input		HF Input *mg*	HF Conc *ppm by volume*	HF Output *mg*	Collection Efficiency %
							Volume *mls*	Conc *mg/ml*				
18	3.3	29.76	62.39	558	59.41	0.99	46.2	1.11	51.3	38.7	50.1	97.7
19	3.3	29.76	64.00	571	59.56	0.99	40.5	1.11	45.0	33.8	44.6	99.1
20	3.2	29.65	62.35	550	60.00	1.00	40.0	1.11	44.4	33.2	38.8	87.4
21	3.2	29.65	62.36	561	58.83	0.98	48.0	0.84	40.3	30.7	36.9	91.6
22	2.0	29.65	49.77	566	46.40	0.77	35.8	1.76	63.0	60.8	62.4	99.0
23	2.0	29.65	51.42	568	47.77	0.80	38.1	1.76	67.1	62.9	67.4	100.4
24	2.0	29.65	51.00	571	47.13	0.79	36.4	1.76	64.1	60.9	63.7	99.4
25	3.0	29.66	59.76	548	57.71	0.96	47.0	1.76	82.7	64.2	84.3	101.9
26	3.0	29.66	61.14	558	57.98	0.97	47.0	1.76	82.7	63.9	81.5	98.5
27	3.0	29.66	61.29	563	57.61	0.96	47.0	1.76	82.7	64.3	86.1	104.1
28	2.0	29.66	51.10	563	47.91	0.80	36.0	3.04	109.4	102.3	110.1	100.6
29	2.0	29.56	50.75	561	47.59	0.79	36.3	3.04	110.4	103.9	109.8	99.5
30	2.0	29.58	50.59	560	47.56	0.79	36.0	3.04	109.4	103.1	110.7	101.2
31	3.0	29.67	59.38	551	57.05	0.95	47.0	3.04	142.9	112.2	150.8	105.5
32	3.0	29.76	60.85	560	57.52	0.96	47.8	3.04	145.3	113.2	148.4	102.1
33	3.1	29.67	61.13	561	57.70	0.96	47.0	3.04	142.9	111.0	151.4	105.9

Attachment B

SAMPLING AND ANALYTICAL DATA, SPECFIIC ION ELECTRODE METHOD WITHOUT DISTILLATION

Run Number	Orifice Pressure *in H2O*	Meter Pressure *in Hg*	Volume of Gas Metered *acf*	Meter Temp. ^{o}R	Volume of Gas Metered *dscf*	Gas Flow Rate *dscfm*	HF Solution Input		HF Input *mg*	HF Conc *ppm by volume*	HF Output *mg*	Collection Efficiency %
							Volume *mls*	Conc *mg/ml*				
Blank,1	3.7	29.53	59.84	574	55.02	0.92	40.0	0.00	0.00	0.00	0.0	-
2	1.6	29.50	44.05	557	41.48	0.69	30.0	0.18	5.40	5.83	4.86	90.0
3	1.6	29.63	44.87	557	42.44	0.71	30.0	0.18	5.40	5.70	5.43	100.6
4	1.6	29.63	44.01	559	41.48	0.69	30.0	0.18	5.40	5.83	5.45	100.9
5	1.6	29.63	44.93	563	42.04	0.70	30.1	0.18	5.42	5.78	5.34	98.5
6	1.6	29.68	45.08	558	42.63	0.71	31.6	0.18	5.69	5.98	6.57	115.5
7	1.6	29.70	44.05	551	42.22	0.70	30.0	0.18	5.40	5.73	5.45	100.9
8	2.9	29.70	61.42	560	58.10	0.97	40.0	0.18	7.20	5.55	7.43	103.2
9	2.9	29.70	61.62	565	57.78	0.96	40.0	0.18	7.20	5.58	7.03	97.6
10	2.9	29.58	60.36	555	57.38	0.96	40.0	0.18	7.20	5.62	6.87	95.4
11	2.9	29.58	61.04	562	57.31	0.96	46.6	0.18	8.39	6.56	7.67	91.4
12	2.9	29.58	62.06	565	57.96	0.97	40.0	0.18	7.20	5.57	7.35	102.1
13	2.9	29.58	61.07	567	56.83	0.95	40.0	0.18	7.20	5.68	6.84	95.0
14	2.4	29.83	54.56	555	52.24	0.87	30.3	1.11	33.6	28.8	34.6	103.0
15	2.4	29.83	55.25	565	51.97	0.87	30.1	1.11	33.4	28.8	33.2	99.4
16	2.1	29.83	51.45	567	48.19	0.80	30.0	1.11	33.3	31.4	32.7	98.2
17	2.0	29.76	48.48	552	46.52	0.78	30.1	1.11	33.4	32.2	33.1	99.1

24.

Attachment B, Continued

Run Number	Orifice Pressure *in H20*	Meter Pressure *in Hg*	Volume of Gas Metered *acf*	Meter Temp. *°R*	Volume of Gas Metered *dscf*	Gas Flow Rate *dscfm*	HF Solution Input		HF Input *mg*	HF Conc *ppm by volume*	HF Output *mg*	Collection Efficiency %
							Volume *ml*	Conc *mg/ml*				
18	3.3	29.76	62.39	558	59.41	0.99	46.2	1.11	51.3	38.7	50.7	98.8
19	3.3	29.76	64.00	571	59.56	0.99	40.5	1.11	45.0	33.8	44.3	98.4
20	3.2	29.65	62.35	550	60.00	1.00	40.00	1.11	44.4	33.2	41.3	93.0
21	3.2	29.65	62.36	561	58.83	0.98	48.0	0.84	40.3	30.7	39.5	98.0
22	2.0	29.65	49.77	566	46.40	0.77	35.8	1.76	63.0	60.8	59.9	95.1
23	2.0	29.65	51.42	568	47.77	0.80	38.1	1.76	67.1	62.9	63.3	94.3
24	2.0	29.65	51.00	571	47.13	0.79	36.4	1.76	64.1	60.9	60.8	94.9
25	3.0	29.66	59.76	548	57.71	0.96	47.0	1.76	82.7	64.2	78.3	94.7
26	3.0	29.66	61.14	558	57.98	0.97	47.0	1.76	82.7	63.9	78.8	95.3
27	3.0	29.66	61.29	563	57.61	0.96	47.0	1.76	82.7	64.3	78.1	94.4
28	2.0	29.66	51.10	563	47.91	0.80	36.0	3.04	109.4	102.3	107.1	97.9
29	2.0	29.56	50.75	561	47.59	0.79	36.3	3.04	110.4	103.9	106.4	96.4
30	2.0	29.58	50.59	560	47.56	0.79	36.0	3.04	109.4	103.1	104.9	95.9
31	3.0	29.67	59.38	551	57.05	0.95	47.0	3.04	142.9	112.2	135.4	94.8
32	3.0	29.67	60.85	560	57.52	0.96	47.8	3.04	145.3	113.2	137.3	94.5
33	3.1	29.67	61.13	561	57.70	0.96	47.0	3.04	142.9	111.0	135.8	95.0